

Lipids, pots and food processing at Hočevarica, Ljubljansko barje, Slovenia

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ABSTRACT – The paper presents the results of lipid analyses of pottery samples from Hočevarica (Ljubljansko barje, Slovenia). Total lipid extracts were subjected to high temperature gas chromatography (HT-GC), gas chromatography-mass spectrometry (GC-MS) and gas chromatography-combustion-isotope ratio mass spectrometry (GC-C-IRMS). The results show that some vessels were used for preparing ruminant meat and vegetable, but also the remains of aquatic food were identified. The processing of non-ruminant meat was detected in a few samples. A high number of pottery samples yielded the presence of beeswax lipids. The charred residual on pottery was AMS ¹⁴C dated.

IZVLEČEK – V članku predstavljamo rezultate analiz lipidov ohranjenih v keramičnem zbiru s Hočevarice. Lipide, ekstrahirane iz ostankov keramičnih posod, smo analizirali s pomočjo plinske kromatografije pri visokih temperaturah (HT-GC), plinske kromatografije sklopljene z masno spektrometrijo (GC-MS) in plinske kromatografije sklopljene z masnim pektrometrom za analizo stabilnih izotopov lahkih elementov preko sežigne enote (GC-C-IRMS). Rezultati kažejo, da so v posodah pripravljali hrano iz mesa prežvekovalcev in zelenjave; redko iz mesa neprežvekovalcev. V drugih so pripravljali hrano iz sladkovodnih rib. V številnih posodah je bil odkrit čebelji vosek. Karbonizirani ostanki na posodah so bili AMS ¹⁴C datirani.

KEY WORDS – lipid analysis; ¹⁴C dates; pottery; Eneolithic; Ljubljansko barje

Introduction

Hočevarica is located at the outfall of Hočevarica drainage channel into the Ljubljanica River between Blatna Brezovica and Verd on the western part of the Ljubljansko barje area (Fig. 1). A small trench (8m²) was excavated in 1998 (Velušček 2004a). The site was recognised as a pile-dwelling settlement embedded in the time span 3650–3520 calBC (for wood samples) (Čufar, Kromer 2004:283) and 3640–3530 calBC (for short-lived seed and carbonised grain samples) (Jeraj 2004:59).

The site stratigraphy consists of ten layers (Fig. 2). While some are of geological provenance, layers 4–8 relate to settling and can be associated with two settlement phases (Velušček 2004b:37–40; 2004c:213–

217). Patches of burnt clay and daub (e.g., house remains) are deposited in well-defined stratigraphic superposition; they correlate with the distribution of vertical wooden piles, and depositions of pottery, stone and wooden tools within the stratified settlements' layers (*ibid.* 40–47).

Palaeobotany and archaeozoology

More than 30000 remains of seeds and fruits of cultivated and gathered wild plants have been found in both settlement contexts. While cereal grains were carbonised, most of the remaining plant remains were unburned. The grains of cultivated *Hordeum vulgare* (six-rowed barley), *Triticum monococcum*

(einkorn wheat) and *Triticum dicoccum* (*T. turgidum* ssp. *Dicoccum*, emmer wheat) were identified; the most abundant cereal at Hočevarica is barley.

However, the remains of wild nuts, fruits and seeds predominated in the archaeobotanical assemblage, comprising *Quercus* sp. *Cupulae* (acorn), *Corylus avellana* (hazelnut), *Malus sylvestris* (crab apple), *Prunus avium* (wild cherry), *Cornus mas* (Cornelian cherry), *Cornus sanguinea* (common dogwood), *Prunus spinosa* (blackthorn), *Rubus fruticosus* (blackberry), *Fragaria vesca* (wild strawberry), *Physalis alkekengi* (winter cherry), and *Trapa natans* (water chestnut). Along with *Papaver somniferum* (opium poppy) seeds, the only remains of an oily plant, *Chenopodium album* (goosefoot), which has seeds rich in oil and starch, were also gathered. Pulses such as *Lathyrus sativus* (grass pea) and *Vicia* sp. (*Vitis vinifera* ssp. *Sylvestris*, wild grapevine) were found in small numbers in the 1st settlement phase (Jeraj 2004:58–59; 2009:79–82).

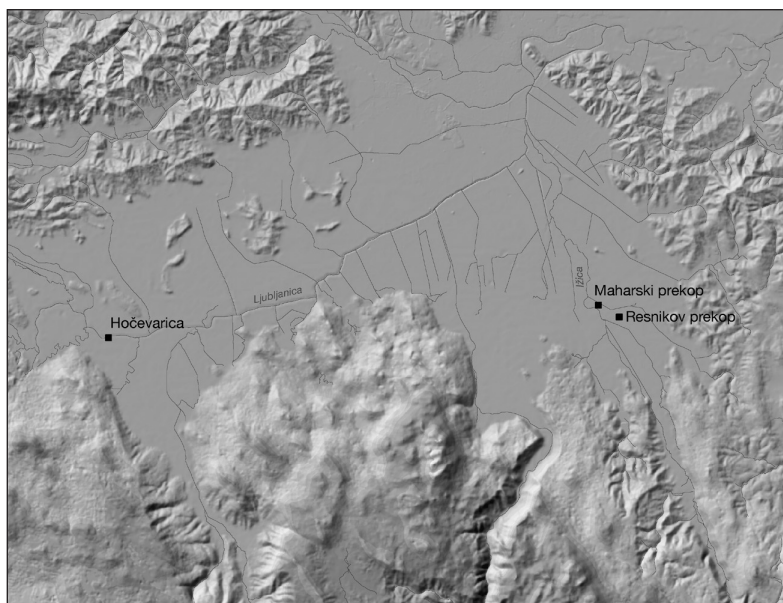


Fig. 1. Map of Ljubljansko barje region with the position of the site at Hočevarica.

It was suggested that while cereals were cultivated in fields situated on moist to damp soils close to the settlement, wild nuts, fruits and seeds were collected along the forest edges and in clearings around the settlement. The water plants were collected in small and shallow meso- to eutrophic lakes which warm up in summer. All the wild plants have been processed in settlement contexts (Tolar et al. 2011:216).

Lab code	Material	Phase	¹⁴ C Conventional age BP	Calibrated date calBC (2σ)	Reference
Hd-18976	wood		4822±39	3695–3521	Čufar, Kromer 2004.Tab. 6.3.2
Hd-22139	wood		4867±26	3702–3636	Čufar et al. 2010.Tab. 4*
Hd-20765	wood		4748±26	3636–3382	Čufar et al. 2010.Tab. 4*
Hd-22305	wood		4825±25	3656–3530	Čufar et al. 2010.Tab. 4*
?	organic sediment		4780±40	3648–3383	Jeraj 2004.62**
??	seed	2	4780±40	3648–3383	Jeraj 2004.59
???	grain	1	4810±40	3691–3518	Jeraj 2004.59
Beta-391181	food residue	2	4910±30	3763–3642	
Beta-391176	food residue	1	4860±30	3704–3539	
Beta-391182	food residue	2	4770±30	3641–3519	
Beta-391178	bos taurus	1	4760±30	3641–3384	
Beta-391183	ovis/capra	2	4740±30	3639–3379	
Beta-391185	Cornus stone	2	4720±30	3635–3376	
Beta-391180	Cornus stone	1	4680±30	3623–3370	
Beta-391177	food residue	1	4780±30	3635–3531	

Tab. 1. Radiocarbon dates from Hočevarica. Dates marked with an asterisk (*) are inconsistently published (Hd-22139 as 4972±25 and Hd-20765 as 4746±26 in Čufar, Kromer 2004). Date for organic sediment (marked by **) by Jeraj (2004) is the same age as date for seed in phase 2. Since Jeraj does not cite lab codes for dates, it is possible that both are the same sample.

The animal bone assemblage consists of 4352 animal remains. About a third of them are fishes and birds, the remainder (63.4%) are mammals. The mammal bones (2757 total) are from at least 14 species (Toškan, Dirjec 2004:76–132). Roe deer (*Capreolus capreolus*) remains predominate, comprising a good third of the mammalian assemblage; the second most frequent was pig/wild boar (*Sus* sp.), accounting for one third. Other species were less frequent. Only red deer (*Cervus elaphus*), beaver (*Castor fiber*), dog (*Canis familiaris*), and the remains of sheep and goat (*Ovis* s. *Capra*) exceeded 5% of all finds. While *Sus scrofa/domesticus* bones

predominate (38.2%) in the 1st settlement phase, *Ovis s. Capra* remains are the most frequent (19.7%) in the 2nd phase (*ibid.* 80).

The evidence of animal slaughter and further meat processing at the site are weak. The proportion of bones with cut and chop marks and/or traces of boiling or roasting was below 10%. However, the analysis of tooth wear showed that most of the pigs were slaughtered in the autumn at an assessed age of 17 to 22 months, and during winter or in early spring, at a probable age of 22 to 27 months (*Toškan, Dirjec 2004.121*). The fish remains consist of five species: common carp, rudd, pike, perch and roach. The carp and rudd remains predominate (*Govedič 2004.133–151*).

Chronology

The Hočevarica radiocarbon sequence is comprised of 13 AMS radiocarbon dates. In addition to the series of four dates on wooden piles used to anchor the dendrochronological sequence and two dates obtained on short-lived botanical samples, an additional two AMS radiocarbon dates from animal bones, two AMS dates on short-lived botanical samples and four dates of carbonised food residues on pottery were obtained recently (Tab. 1).

Complementary samples allow a better understanding of the chronology of activities at the site. The radiocarbon dates of bones and carbonised food/organic residues on pottery date events relating to the preparation and disposal of food, and thus complement the dates of the wooden structures relating to building and construction events. The floating oak chronology of 139 years from Hočevarica (HOC-QUSP1) is dated between 3685 and 3547 (± 10) BC, which suggests an end to building activities after around 3550 BC (*Čufar et al. 2010*).

On the other hand, the majority of AMS dates on short-lived samples concentrate between 3630–3350 calBC (Fig. 3). The wide spread of values can be attributed to a wiggles in the calibration curve between 3620–3520 and 3480–3380 calBC. However, it seems that activities at the site reflected in the short-lived sam-

ples began well before the end of the building activities, before 3600 calBC, and continued for a few decades after building activities had ended. This long span of activities corresponds well with the two settlement phases.

Two dates on charred food residues on pottery are older than the oldest dates on the wooden piles. Lipid analysis on one sample (Beta-391176) from the first phase yielded a lipid concentration high enough (01HO; Tab. 2) to suggest that the pot was used to cook a ruminant/plant mixture. The concentration of lipids in the other sample (Beta-391181, 18HO; Tab. 2) was too low to allow a determination of food-stuffs. However, as this sample is associated with the second phase, it appears too old. At the moment, we have no dates on fish bones or food residues associated with aquatic foodstuffs that would demonstrate the presence of a reservoir effect. Therefore, both early dates could suggest earlier activities at the site or a reservoir effect.

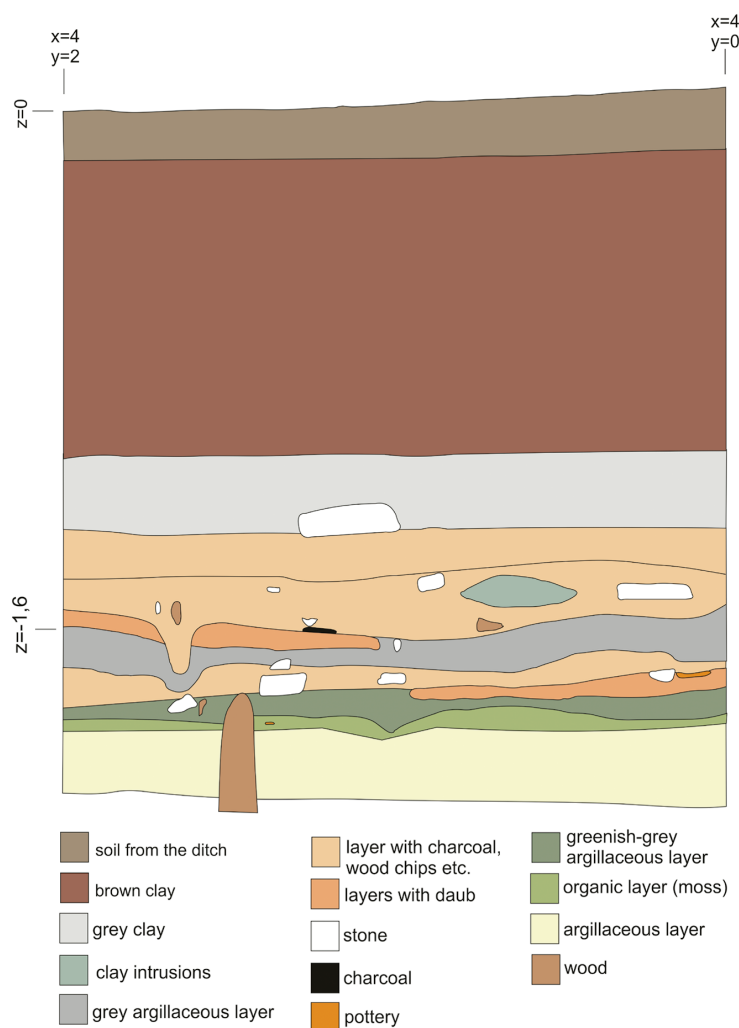


Fig. 2. Northern cross-section of the trench at Hočevarica (after Velušček 2004.Fig. 3.1.5).

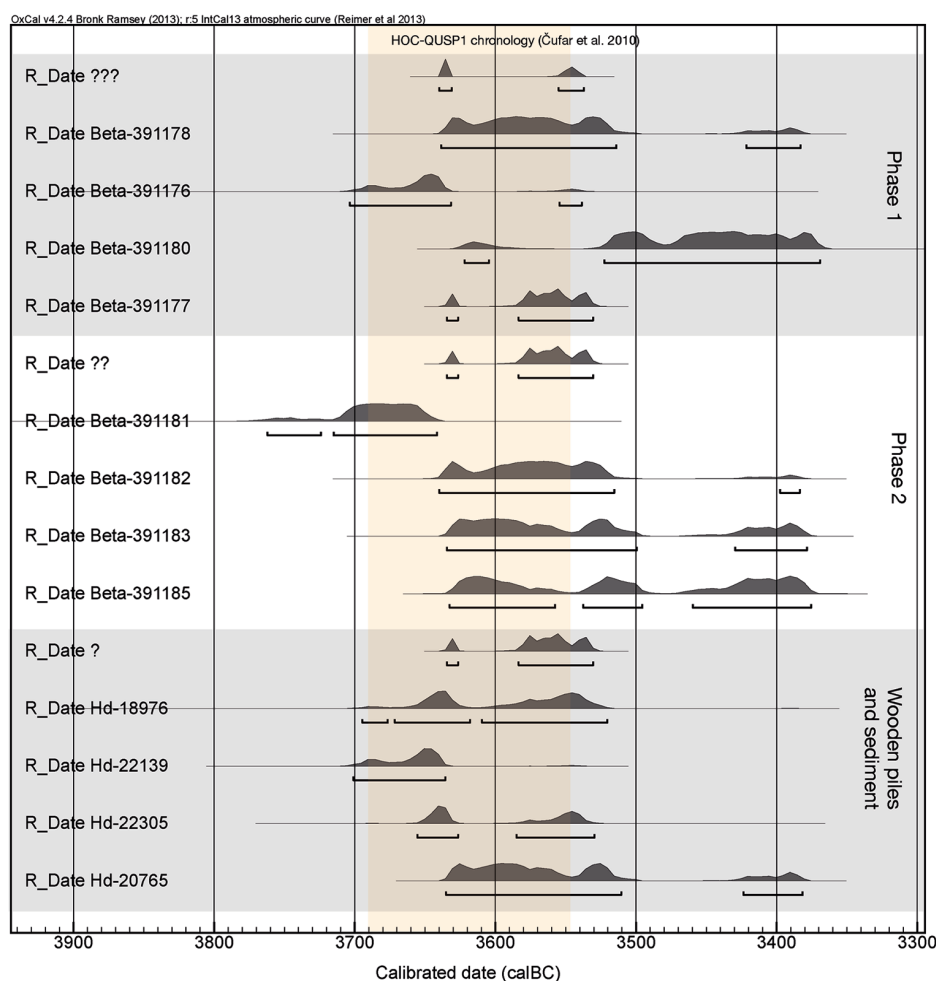


Fig. 3. Calibrated radiocarbon dates from Hočevarica in relation to the HOC-QUSP1 chronology.

These new dates suggest a long and complex chronological sequence for the Hočevarica site. It appears that the site was settled for almost 200 years, had two distinct phases of occupation, and shows possible evidence of activities before the wooden structures were built.

The pottery

For the present study, we analysed 35 pottery samples from Hočevarica by hand lens to identify inclusions, their size and frequency, and the presence of voids. The samples were chosen on the basis of typology (see *Velušček 2004d.169–212*) and on the basis of the presence of charred food remains on the interior surface of the vessels. Most of the samples came from fragments of vessel rims and walls; only 9 samples were attributed to types according to their morphology: 3 pots, 4 dishes, and 2 bowls (Fig. 4; Tab. 2).

The vessel types are similar to the pottery assemblage from the contemporary site at Maharski pre-

kop in the south-eastern part of Ljubljansko barje (*Bregant 1974a; 1974b; 1975; Velušček 2004d.184–212*). The majority of the vessels can be attributed to various types of pots (*Velušček 2004d.186–194*) and dishes (*ibid. 196–203*), but other forms are also present (cups, miniature vessels, hanging vessels and other special forms; *ibid. 195, 203*).

Similarly, the technological characteristics of the Hočevarica pottery assemblage are comparable to vessels from Maharski prekop (*Žibrat Gašparič 2013.153–155*). The vessels are primarily dark grey, brown and black, and most were fired in a reducing atmosphere. Most of the pottery is poorly made and prone to mechanical decomposition; only the decorated vessels are of better quality and have polished surfaces or slips applied to the surface (*Velušček 2004d.184–185*).

We could divide the pottery samples into two technological groups according to their inclusions (descriptions after *Horvat 1999*): most of the samples have calcite/limestone inclusions (82.8%), while the

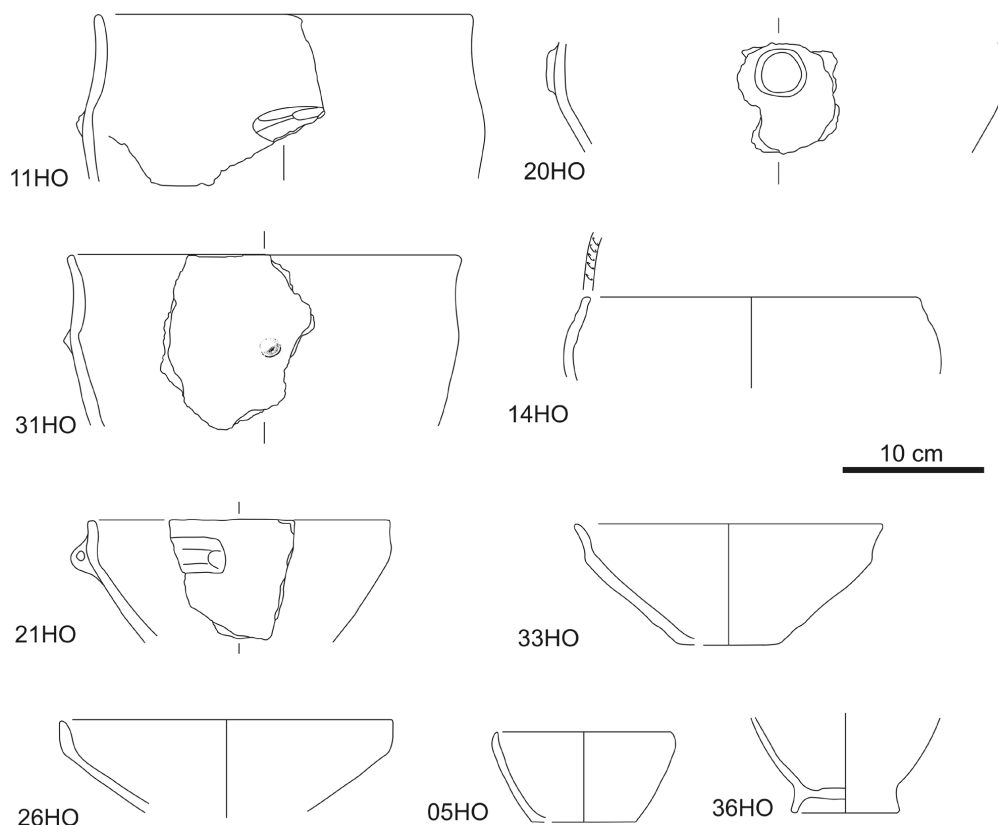


Fig. 4. Pottery samples bearing traces of ruminant fat (14HO, 33HO), mixed animal fats (05HO), mixed animal and plant fats (31HO), mixed animal fats and beeswax (21HO, 26HO, 36HO), freshwater animal oils (11HO) and a mixture of dairy fat and beeswax (20HO) (drawings after Velušček 2004a.169–183).

remainder are made of non-calcareous clay and have only quartz inclusions (17.2%). In the group with quartz, most of the inclusions comprise very fine (less than 0.25mm) or medium-size sand (0.25 to 0.50mm). Most of the samples with calcite/limestone have medium-size sand inclusions (0.25 to 0.50mm), but coarse sand is present (0.50 to 2.00mm) in a third of the samples.

The pottery samples from Hočevarica have voids, usually on both surfaces, in the size of medium to coarse sand fraction, and many have an angular shape similar to calcite crystals. This could be the result of calcite dissolved from the vessels. Such chemical changes in pottery are common post-depositional processes (Rice 1987.421). A similar situation could be observed at the contemporary site at Krašnja near Lukovica (Žibrat Gašparič et al. 2014).

All the pottery samples were handmade and their surfaces burnished; smoothing and polishing were also present. One of the vessels (10HO) was decorated with a grey-black slip on both the interior and exterior surfaces. They were fired in an incomplete oxidising (51.4%) and a reducing atmosphere (34.3%), while the other samples were fired in a reducing at-

mosphere with an oxidising atmosphere at the end of firing.

The pottery from the calcite/limestone group at Hočevarica has characteristics very similar to fabric MP-1 from Maharski prekop, which is a non-calcareous clay with frequent calcite grains added as temper and is the most common fabric found at that site (Žibrat Gašparič 2013.154). On the other hand, the group with quartz inclusions from Hočevarica differs from the fabrics described at Maharski prekop and could display a new technology in the later phase of the settlement, since the samples of the quartz group all come from the 2nd settlement phase at Hočevarica. This hypothesis would have to be tested with additional pottery samples, as well as with a petrographical analysis of thin sections.

Materials and methods

A total of 36 selected pottery samples were first cleaned to remove exogenous lipids, and then ground to a fine powder. For lipid extraction, about 2g of sample were transferred to a 50ml vial and 20µl of internal standard (*n*-tetratriacontane, 1mg/mL in *n*-hexane) were added. Lipids were ultrasonically ex-

tracted with a mixture of methanol and chloroform (1:2 v/v, 24mL, 2 x 30min). The solvent extract was removed into a glass flask and reduced to a small volume by rotary evaporation. The residue of solvent extract was transferred to a 2ml glass vial and evaporated to dryness under a gentle stream of nitrogen to obtain the total lipid extract (TLE). The aliquot (500µl) of the TLE was treated with BSTFA (N, O-bis(trimethylsilyl)-trifluoroacetamide, 40µl; 70°C, 60min), evaporated to dryness and re-dissolved in *n*-hexane. The resulting trimethylsilyl derivatives were analysed using high-temperature gas chromatography (HT-GC) and, where necessary, combined GC-MS analyses were performed to identify the structure of the components (Evershed et al. 1990). All HT-GC analyses were performed on Agilent Technology 6890N GC system equipment with DB-5HT capillary column (15m x 0.32m x 0.10µm). Temperature program: initial temperature 50°C (1min), increasing to 350°C (10 min) at a rate of 10°C/min. Helium was used as a carrier gas and a flame ionisation detector to monitor the column effluent.

Another aliquot (500µL) of solvent extract was used to prepare free fatty acids methyl esters (FAMES) by adding 100µL of BF₃-methanol (14% w/v, Sigma Aldrich, 70°C, 60min). The methyl derivatives were extracted with *n*-hexane and analysed by GC-MS and GC-C-IRMS using standard protocols (Evershed et al. 1994; Mottram et al. 1999; Greg, Slater 2010; Ogrinc et al. 2012). For GC-C-IRMS (Isoprime GV system, Micromass, Manchester, UK) the accuracy of repeated measurements was ±0.3‰.

In addition, powder samples (~1mg) were analysed by elemental analysis isotope ratio mass spectrometry (IRMS) as previously reported (Ogrinc et al. 2012; Budja et al. 2013). Stable isotope results are expressed as δ¹³C or δ¹⁵N values in per mil (‰) relative to the VPDB and AIR international standard, respectively. The accuracy of measurements was ±0.2‰ for δ¹³C and ±0.3‰ for δ¹⁵N.

Results and discussion

The average and standard deviations from bulk potsherd samples are -28.3±1.6‰ and +4.5±2.0‰ for δ¹³C and δ¹⁵N, respectively (Fig. 5; Tab. 2). These data fall in the range expected for C₃ plant and degraded animal tissues whose subsistence was based mainly on C₃ plants. The δ¹⁵N values of terrestrial plant proteins are around +3‰, while proteins derived from terrestrial herbivores from temperate Europe should not exceed δ¹⁵N values of +7.0‰ (Richards et al. 2003), although protein derived from domestic animals (such as pigs) may be higher (Privat et al. 2002; Polet, Katzenberg 2003; Richards et al. 2003; Ogrinc, Budja 2005). At Hočevarica, only three samples (01HO, 03HO and 06HO) have δ¹⁵N values higher than +7.0‰. Thus the variations in the carbon and nitrogen isotope ratios in our sample show that a wide diversity of animal and plant food was processed in the vessels. No sample has an δ¹⁵N value greater than 9‰ consistent with processing aquatic products with a high trophic level (Fig. 5). However, data on fish species from modern and archaeological samples from lacustrine environments demonstrates a wide range of nitrogen values due to the diverse mixture of aquatic food sources. For example, the δ¹⁵N values of freshwater fish in Lake Baikal range from +7.3 to +13.7‰ (Katzenberg, Weber 1999). And Melanie J. Miller et al. (2010) reported that the modern fish δ¹⁵N values of Lake Titicaca range from +4.1 to +9.5‰, while the majority of the δ¹⁵N values in archaeological fish samples ranged from +5.1 to +7.7‰.

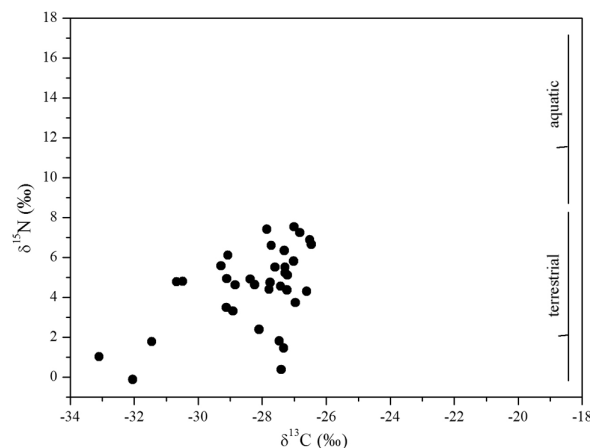


Fig. 5. Bulk stable isotope values of pottery samples from Hočevarica. The vertical bars show 95% confidence intervals and the median stable nitrogen isotope value from literature data.

In order to obtain more reliable information on the processing of different commodities in pottery vessels from Hočevarica, more specific chemical and molecular analysis, including lipid analysis, were performed. Lipid preservation in our samples was very good, with more than 75% of potsherds containing appreciable quantities of lipid (Tab. 2).

Lipid biomarkers

Even-carbon number *n*-alkanoic acids that range from C_{12:0} to C_{22:0} were observed in analysed sherds (Fig. 6). In addition, monounsaturated fatty acids

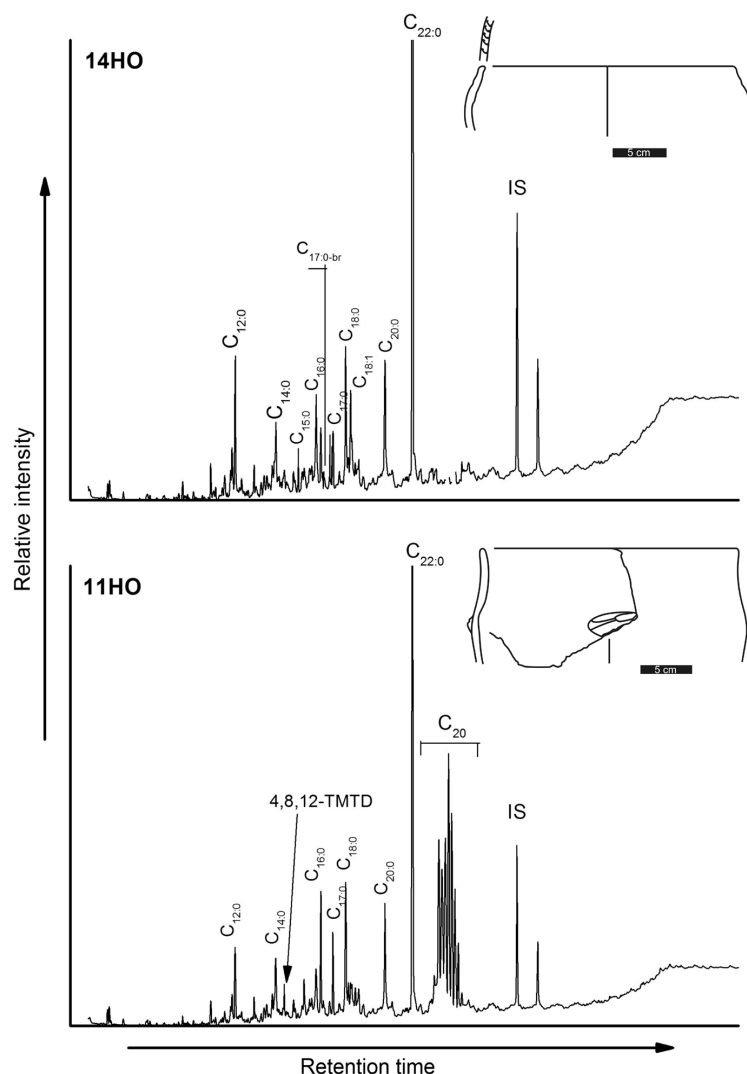


Fig. 6. The representative GC-MS total ion chromatograms of the fatty acids methyl esters (FAMES) with different $C_{16:0}$ and $C_{18:0}$ abundance extracted from the Hočevarica pottery samples 11HO and 14HO.

$C_{18:1}$ were present in the lipid extracts of all samples (Tab. 1). The presence of odd number ($C_{15:0}$ and $C_{17:0}$) and/or a low amount of branched chain of $C_{17:0}$ was determined in 50% of the pottery samples (02HO, 05HO, 14HO, 20HO, 21HO, 22HO, 25HO, 26HO, 27HO, 31HO, 33HO, 34HO, 36HO). The presence of these acids together with two double bonds positional isomers of $C_{18:1}$ indicates ruminant animal fats that have been biosynthesised in the gut and rumen (Dudd et al. 1999; Regert 2011). The parallel biomarkers, *i.e.* triacylglycerols (TAGs) and their degradation products (diacylglycerols (DAGs) and monoacylglycerols (MAGs) were detected in 9 sherds (02HO, 05HO, 14HO, 20HO, 21HO, 26HO, 27HO, 34HO, 36HO), confirming the presence of degraded animal fats (Tab. 2; Fig. 7). However, the TAG distribution could be identified in three sherds (20HO, 21HO and 26HO), while in the remaining samples

only traces of TAGs were observed. The narrow distribution of TAGs in these three sherds, ranging from C_{42} to C_{52} , indicates the presence of ruminant adipose or dairy fats.

The presence of saturated and mono-saturated fatty acids in a range from C_{20} to C_{24} , together with a high proportion of $C_{16:0}$ and minor amounts of $C_{12:0}$ and $C_{18:0}$ acids are indicative of aquatic oils and thus provide evidence that freshwater foods were processed in these vessels (Hansel et al. 2004; Craig et al. 2011; 2013; Cramp et al. 2014). Such a lipid profile was observed in 35% of the samples (04HO, 06HO, 09HO, 10HO, 11HO, 12HO, 13HO, 17HO and 18HO). In addition, in these samples 4,8,12-trimethyltridecanoic acid (4,8,12-TMDT) at low concentrations was also identified. This component is a characteristic lipid biomarker of aquatic resources (Hansel et al. 2004) (Fig. 6).

Alongside the identification of animal or aquatic fats, a high percentage of samples (81%) yielded the presence of beeswax lipids (Tab. 2). In five samples (20HO, 21HO, 25HO, 26HO, 36HO) the lipid distribution indicate the high content of degraded beeswax lipids, while in other samples only traces of wax lipids are present. Beeswax lipids may indicate the addition of honey to other foodstuffs or the application of beeswax to pottery vessels to improve impermeability (Regert et al. 2001; Kimpe et al. 2002; Copley et al. 2005). Although in most of the samples only trace levels of this particular commodity were detected, its presence indicates that beeswax was utilised at Hočevarica in pottery vessels associated with cooking/processing foodstuffs or applied as a coating.

Long-chain ketones (C_{31} , C_{33} and C_{35}) were observed in most samples with preserved lipids, except in 05HO. Long-chain ketones have been widely reported as components of the epicuticular waxes of higher plants (Walton 1990), but can be also formed from the condensation of fatty acids ($C_{16:0}$ and $C_{18:0}$) during the heating of vessels to temperatures in excess of 400°C (Evershed et al. 1999). The presence of long-chain ketones together with thermally pro-

Lab. sample no.	ID. No.	Context	¹⁴ C Lab. no.	¹⁴ C conv. age BP	Fabric group	Description	TLE (μg g ⁻¹)	δ ¹³ C bulk (‰)	δ ¹⁵ N bulk (‰)	δ ¹³ C _{16:0} (‰)	δ ¹³ C _{18:0} (‰)
01HO	126	phase 1	Beta-391176	4860±30	calcite	vessel wall	36.5	-26.8	7.2	-29.0	-29.1
02HO	165	phase 1	Beta-391177	4780±30	calcite	vessel wall	25.3	-29.1	4.9	-28.0	-29.2
03HO	073	phase 1			calcite	vessel wall	48.3	-27.0	7.5	n/d	n/d
04HO	075	phase 1			calcite	vessel rim with wall	32.9	-27.7	6.6	-31.0	-25.7
05HO	080	phase 1			calcite	dish	39.0	-27.3	5.5	-27.8	-27.4
06HO	135	phase 1			calcite	vessel wall	96.5	-27.9	7.4	-31.1	-27.3
07HO	138	phase 1			calcite	vessel wall	42.7	-32.0	-0.1	-34.3	-29.0
08HO	174	phase 1			calcite	vessel rim with wall	40.8	-27.4	0.4	-29.8	-28.2
09HO	087	phase 1			calcite	vessel rim with wall	13.1	-31.5	1.8	-30.7	-27.1
10HO	076	phase 1			calcite	vessel rim with wall	10.9	-27.8	4.7	-32.2	-28.5
11HO	PN0081	phase 1			calcite	pot	71.3	-29.1	3.5	-29.8	-27.7
12HO	068	phase 1/2			calcite	vessel rim with wall	78.6	-26.5	6.9	-30.7	-28.5
13HO	067	phase 1/2			calcite	vessel wall	37.8	-27.0	3.7	-32.4	-27.6
14HO	PN0135	phase 1/2			calcite	pot	51.5	-27.5	1.8	-25.5	-27.9
16HO	049	phase 1/2			calcite	vessel wall	108	-27.3	1.5	-36.0	-29.9
17HO	082	phase 2			calcite	vessel base with wall	27.8	-26.6	4.3	-31.5	-28.8
18HO	088	phase 2	Beta-391181	4910±30	calcite	vessel wall	5.9	-28.4	4.9	n/d	n/d
19HO	029	phase 2			calcite	vessel wall	3.1	-27.8	4.4	n/d	n/d
20HO	032	phase 2			quartz	pot?	211	-30.7	4.8	-27.3	-33.9
21HO	PN0049	phase 2			quartz	dish	63.3	-30.5	4.8	-26.7	-28.5
22HO	035	phase 2	Beta-391182	4770±30	calcite	vessel wall	29.2	-27.3	6.3	-29.8	-29.1
23HO	020	phase 2			calcite	vessel wall	23.6	-27.0	5.8	-30.6	-26.8
24HO	017	phase 2			quartz	vessel rim with wall	2.1	-27.6	5.5	n/d	n/d
25HO	169	phase 2			quartz	vessel wall	73.9	-27.2	5.1	-26.5	-28.4
26HO	025	phase 2			quartz	dish	53.3	-27.3	5.2	-28.4	-29.2
27HO	019	phase 2			calcite	vessel wall	15.9	-27.2	4.4	-30.3	-31.4
28HO	120	phase 1			calcite	vessel wall	1.6	-28.2	4.6	n/d	n/d
29HO	121	phase 1			calcite	vessel wall	6.0	-29.3	5.6	n/d	n/d
30HO	089	phase 1			calcite	bowl	4.7	-26.5	6.7	n/d	n/d
31HO	085	phase 1			calcite	pot	26.4	-28.8	4.6	-28.6	-28.2
32HO	078	phase 1			calcite	vessel wall	18.3	-29.1	6.1	n/d	n/d
33HO	061	planum 4/4			calcite	dish	12.4	-27.4	4.6	-28.1	-29.5
34HO	008	SU 4/7			quartz	vessel wall	7.0	-28.9	3.3	-26.3	-27.2
35HO	003	SU 1/2			calcite	vessel wall	6.9	-33.1	1.0	n/d	n/d
36HO	PN0138	E cross-section			calcite	bowl?	6.2	-28.1	2.4	-28.2	-29.1

Tab. 2. A summary of the organic residues detected in pottery samples from Hočevarica, Ljubljansko barje region. Key: MAG – moniacylglycerols; DAG – diacylglycerols; TAG – triacylglycerols; A – n-alkanes; OH – n-alcohols; K – ketones; WE – wax esters; (tr) – trace; n/d – not detected.

$\Delta^{13}\text{C}$ (‰)	$\text{C}_{16:0}/\text{C}_{18:0}$	Fatty Acids (FA)	Other lipids	Predominant commodity type	Reference
0.0	1.48	$\text{C}_{12:0}$, $\text{C}_{14:0}$, $\text{C}_{16:0}$, $\text{C}_{17:1}$, $\text{C}_{18:1}$, $\text{C}_{18:0}$, $\text{C}_{20:0}$, $\text{C}_{22:0}$	K, WE	mixture ruminant, plant	Not published
-1.2	1.45	$\text{C}_{12:0}$, $\text{C}_{14:0}$, $\text{C}_{15:0}$, $\text{C}_{16:0}$, $\text{C}_{17:0}$ -br, $\text{C}_{17:0}$, $\text{C}_{18:1}$, $\text{C}_{18:0}$, $\text{C}_{20:0}$, $\text{C}_{22:0}$	K, WE, DAG(tr), TAG(tr)	ruminant	Not published
n/d	n/d	n/d	K	n/d	Not published
5.3	1.50	$\text{C}_{12:0}$, $\text{C}_{14:0}$, TMDT, $\text{C}_{16:0}$, $\text{C}_{17:1}$, $\text{C}_{17:0}$, $\text{C}_{18:1}$, $\text{C}_{18:0}$, $\text{C}_{20:0}$, $\text{C}_{22:0}$	K	freshwater	Not published
0.4	0.74	$\text{C}_{12:0}$, $\text{C}_{14:0}$, $\text{C}_{15:0}$, $\text{C}_{16:0}$, $\text{C}_{17:1}$, $\text{C}_{18:1}$, $\text{C}_{18:0}$, $\text{C}_{20:0}$, $\text{C}_{22:0}$	WE, DAG, TAG	mixture ruminant, non-ruminant	<i>Velušček 2004.Pl. 4.1.5:7</i>
3.8	1.44	$\text{C}_{12:0}$, $\text{C}_{14:0}$, TMDT, $\text{C}_{16:0}$, $\text{C}_{17:1}$, $\text{C}_{17:0}$, $\text{C}_{18:1}$, $\text{C}_{18:0}$, $\text{C}_{20:0}$, $\text{C}_{22:0}$	K, WE(tr)	freshwater	Not published
5.3	2.80	$\text{C}_{12:0}$, $\text{C}_{14:0}$, $\text{C}_{16:0}$, $\text{C}_{18:1}$, $\text{C}_{18:0}$, $\text{C}_{20:0}$, $\text{C}_{22:0}$	K, WE(tr)	non-ruminant	Not published
1.6	0.76	$\text{C}_{12:0}$, $\text{C}_{16:0}$, $\text{C}_{17:1}$, $\text{C}_{18:1}$, $\text{C}_{18:0}$, $\text{C}_{20:0}$, $\text{C}_{22:0}$	K, WE(tr)	non-ruminant	Not published
3.6	3.79	$\text{C}_{12:0}$, $\text{C}_{14:0}$, TMDT, $\text{C}_{16:0}$, $\text{C}_{17:1}$, $\text{C}_{18:1}$, $\text{C}_{18:0}$, $\text{C}_{20:0}$, $\text{C}_{22:0}$	K, WE(tr)	freshwater	Not published
3.7	1.97	$\text{C}_{12:0}$, $\text{C}_{14:0}$, TMDT, $\text{C}_{16:0}$, $\text{C}_{17:1}$, $\text{C}_{18:1}$, $\text{C}_{18:0}$, $\text{C}_{20:0}$, $\text{C}_{22:0}$	K, WE(tr)	freshwater	Not published
2.1	0.94	$\text{C}_{12:0}$, $\text{C}_{14:0}$, TMDT, $\text{C}_{16:0}$, $\text{C}_{17:1}$, $\text{C}_{18:1}$, $\text{C}_{18:0}$, $\text{C}_{20:0}$, $\text{C}_{22:0}$	K, WE(tr)	freshwater	<i>Velušček 2004.Pl. 4.1.3:2</i>
2.2	1.15	$\text{C}_{12:0}$, $\text{C}_{14:0}$, TMDT, $\text{C}_{16:0}$, $\text{C}_{17:1}$, $\text{C}_{18:1}$, $\text{C}_{18:0}$, $\text{C}_{20:0}$, $\text{C}_{22:0}$	K, WE(tr)	freshwater	Not published
4.8	0.74	$\text{C}_{12:0}$, $\text{C}_{14:0}$, TMDT, $\text{C}_{16:0}$, $\text{C}_{17:1}$, $\text{C}_{18:1}$, $\text{C}_{18:0}$, $\text{C}_{20:0}$, $\text{C}_{22:0}$	K, WE(tr)	freshwater	Not published
-2.4	0.74	$\text{C}_{12:0}$, $\text{C}_{14:0}$, $\text{C}_{15:0}$, $\text{C}_{16:0}$, $\text{C}_{17:0}$ -br, $\text{C}_{17:0}$, $\text{C}_{18:1}$, $\text{C}_{18:0}$, $\text{C}_{20:0}$, $\text{C}_{22:0}$	K, WE, TAG(tr)	ruminant	<i>Velušček 2004.Pl. 4.1.6:5</i>
6.1	2.22	$\text{C}_{12:0}$, $\text{C}_{14:0}$, TMDT, $\text{C}_{15:0}$, $\text{C}_{16:0}$, $\text{C}_{17:1}$, $\text{C}_{18:1}$, $\text{C}_{18:0}$, $\text{C}_{20:0}$, $\text{C}_{22:0}$	K, WE(tr)	freshwater	Not published
2.7	1.35	$\text{C}_{12:0}$, $\text{C}_{14:0}$, TMDT, $\text{C}_{15:0}$, $\text{C}_{15:1}$, $\text{C}_{16:0}$, $\text{C}_{16:1}$, $\text{C}_{17:1}$, $\text{C}_{18:1}$, $\text{C}_{18:0}$, $\text{C}_{20:0}$, $\text{C}_{22:0}$	K, WE(tr)	freshwater	Not published
n/d	n/d	n/d	n/d	n/d	Not published
n/d	n/d	n/d	n/d	n/d	Not published
-6.6	1.19	$\text{C}_{12:0}$, $\text{C}_{14:0}$, $\text{C}_{15:0}$, $\text{C}_{16:0}$, $\text{C}_{16:1}$, $\text{C}_{17:0}$ -br, $\text{C}_{17:0}$, $\text{C}_{18:1}$, $\text{C}_{18:0}$, $\text{C}_{21:0}$, $\text{C}_{20:0}$, $\text{C}_{22:0}$, $\text{C}_{24:0}$	A, OH, K, WE, MAG, DAG, TAG	mixture ruminant dairy fats and degraded beeswax	<i>Velušček 2004.Pl. 4.1.9:10</i>
-1.8	1.26	$\text{C}_{12:0}$, $\text{C}_{14:0}$, $\text{C}_{15:0}$, $\text{C}_{16:0}$, $\text{C}_{17:0}$, $\text{C}_{18:1}$, $\text{C}_{18:0}$, $\text{C}_{20:0}$, $\text{C}_{22:0}$, $\text{C}_{24:0}$	A, OH, K, WE, DAG, TAG	mixture ruminant fats and degraded beeswax	<i>Velušček 2004.Pl. 4.1.8:1</i>
0.7	1.22	$\text{C}_{12:0}$, $\text{C}_{14:1}$, $\text{C}_{15:0}$, $\text{C}_{16:0}$, $\text{C}_{17:1}$, $\text{C}_{17:0}$, $\text{C}_{18:1}$, $\text{C}_{18:0}$, $\text{C}_{20:0}$, $\text{C}_{22:0}$	K	mixture ruminant, plant	Not published
3.8	2.02	$\text{C}_{12:0}$, $\text{C}_{14:0}$, $\text{C}_{16:0}$, $\text{C}_{18:1}$, $\text{C}_{18:0}$, $\text{C}_{20:0}$, $\text{C}_{22:0}$	K	non-ruminant	Not published
n/d	n/d	n/d	n/d	n/d	Not published
-1.9	1.93	$\text{C}_{12:0}$, $\text{C}_{14:0}$, $\text{C}_{15:0}$, $\text{C}_{16:0}$, $\text{C}_{17:0}$, $\text{C}_{18:1}$, $\text{C}_{18:0}$, $\text{C}_{20:0}$, $\text{C}_{22:0}$, $\text{C}_{24:0}$	A, OH, K, WE	mixture ruminant fats and degraded beeswax	Not published
-0.8	2.28	$\text{C}_{12:0}$, $\text{C}_{14:0}$, $\text{C}_{15:0}$, $\text{C}_{16:0}$, $\text{C}_{17:0}$, $\text{C}_{18:1}$, $\text{C}_{18:0}$, $\text{C}_{20:0}$, $\text{C}_{22:0}$, $\text{C}_{24:0}$	A, OH, K, WE, MAG, DAG, TAG	mixture ruminant fats and degraded beeswax	<i>Velušček 2004.Pl. 4.1.10:2</i>
-1.1	0.85	$\text{C}_{12:0}$, $\text{C}_{14:0}$, $\text{C}_{15:0}$, $\text{C}_{15:1}$, $\text{C}_{16:0}$, $\text{C}_{17:0}$ -br, $\text{C}_{17:0}$, $\text{C}_{18:1}$, $\text{C}_{18:0}$, $\text{C}_{20:0}$, $\text{C}_{22:0}$	K, DAG(tr), TAG(tr)	ruminant	Not published
n/d	n/d	n/d	n/d	n/d	Not published
n/d	n/d	n/d	n/d	n/d	Not published
n/d	n/d	n/d	n/d	n/d	Not published
0.4	1.11	$\text{C}_{12:0}$, $\text{C}_{14:0}$, $\text{C}_{15:0}$, $\text{C}_{15:1}$, $\text{C}_{16:0}$, $\text{C}_{17:0}$, $\text{C}_{17:1}$, $\text{C}_{18:1}$, $\text{C}_{18:0}$, $\text{C}_{20:0}$, $\text{C}_{22:0}$	K, WE(tr)	mixture ruminant, plant	<i>Velušček 2004.Pl. 4.1.3:3</i>
n/d	n/d	n/d	n/d	n/d	Not published
-1.4	1.67	$\text{C}_{12:0}$, $\text{C}_{14:0}$, $\text{C}_{16:0}$, $\text{C}_{17:0}$ -br, $\text{C}_{17:0}$, $\text{C}_{18:1}$, $\text{C}_{18:0}$, $\text{C}_{20:0}$, $\text{C}_{22:0}$	K	ruminant	<i>Velušček 2004.Pl. 4.1.7:1</i>
-1.0	0.24	$\text{C}_{12:0}$, $\text{C}_{14:0}$, $\text{C}_{15:0}$, $\text{C}_{15:1}$, $\text{C}_{16:0}$, $\text{C}_{16:1}$, $\text{C}_{17:0}$ -br, $\text{C}_{17:0}$, $\text{C}_{18:1}$, $\text{C}_{18:0}$, $\text{C}_{21:0}$, $\text{C}_{20:0}$, $\text{C}_{22:0}$, $\text{C}_{24:0}$	K, TAG, WE(tr)	ruminant	Not published
n/d	n/d	n/d	n/d	n/d	Not published
-0.9	0.81	$\text{C}_{12:0}$, $\text{C}_{14:0}$, $\text{C}_{15:0}$, $\text{C}_{16:0}$, $\text{C}_{17:0}$, $\text{C}_{18:1}$, $\text{C}_{18:0}$, $\text{C}_{20:0}$, $\text{C}_{22:0}$	A, OH, K, WE, DAG(tr), TAG(tr)	mixture ruminant fats and degraded beeswax	<i>Velušček 2004.Pl. 4.1.7:3</i>

duced ω -(*o*-alkylphenyl)alkanoic acids implies that their formation is mainly related to heating to high temperatures.

Stable carbon isotope composition of fatty acids

Further information regarding the source of the organic residues was obtained by measuring the stable carbon isotope ratio of saturated fatty acids $C_{16:0}$ and $C_{18:0}$ preserved in sufficient quantities in the pottery samples. The results were compared with modern reference animal data obtained from the literature presented in Figure 8 (Evershed et al. 2002; Copley et al. 2005; Craig et al. 2007; 2012).

Twelve samples (04HO, 06HO, 07HO, 08HO, 09HO, 10HO, 11HO, 12HO, 13HO, 16HO, 17HO and 23HO) yielded $\delta^{13}C$ values closer to those of lipid extracts from modern pottery vessels used to prepare freshwater and non-ruminant animals (Copley et al. 2005) (Fig. 8). Although nine of them (04HO, 06HO, 09HO, 10HO, 11HO, 12HO, 13HO, 16HO, 17HO) have aquatic biomarkers present, their use cannot be resolved more specifically. Non-ruminant, terrestrial animal contribution/origin could not be excluded, since the animal bone assemblage contains a high percentage of boar/pig (>30%) (Toškan, Dirjec 2004).

35% of samples (02HO, 14HO, 21HO, 25HO, 26HO, 27HO, 33HO, 34HO, 36HO) plot in the range for ruminant adipose fats (Fig. 8). The $C_{16:0}/C_{18:0}$ ratios of fatty acids for these samples range between 0.74 and 2.28 values (Tab. 2) typical of ruminant adipose fat (Copley et al. 2005). The distribution of the data (Fig. 8) and $\delta^{15}N$ values of samples (average value $4.4 \pm 1.2\text{‰}$) suggested that the population at Hočevarica used diverse domesticated (goat, cattle) or wild (deer) animal products in their diet. The sample 20HO plots in the region typical of ruminant dairy fats. The processing of dairy products in this pottery vessel is further supported by the distribution of lipids (Fig. 7).

A further 15% of the samples (01HO, 05HO, 22HO, 31HO) fall close to the limit value between non-ruminant and ruminant fat ($\Delta^{13}C = \delta^{13}C_{18:0} - \delta^{13}C_{16:0} = 0\text{‰}$). However, not all samples could be assigned

to meat mixtures exclusively. In vegetable oils, for example, the $C_{18:1}$ fatty acid is enriched in ^{13}C compared to $C_{18:0}$ (Spangenberg, Ogrinc 2001). A ^{13}C -enrichment of $C_{18:1}$ (up to 2.3‰) compared to $C_{18:0}$ acid was also observed in three pottery vessels (01HO, 22HO and 31HO) suggesting an admixture of plant-animal fats.

Conclusions

The results of stable isotope data and the more specific product identification based on available lipids indicate varied vessel use: pots were used to cook both aquatic and terrestrial products.

The ruminant animal fats of either domestic (cattle, goat) or wild (deer) origin were the most frequently processed products preserved in the Hočevarica pottery samples (Tab. 2; 02HO, 05HO, 14HO, 21HO, 22HO, 25HO, 26HO, 27HO, 31HO, 33HO, 34HO, 36HO). These samples come from all the analysed settlement phases at Hočevarica and display a variety of different types and technologies (both the calcite/limestone group and the quartz group). This confirms that ruminant animal fat was processed in a variety of vessels, such as pots (14HO, 31HO), dishes (21HO, 26HO, 33HO) and bowls (05HO, 36HO) (Fig. 4).

The processing of non-ruminant animal fats was detected in only three samples from Hočevarica that come from both main settlement phases, all made from the most common technological group with added calcite/limestone inclusions (Tab. 2; 07HO, 08HO, 23HO).

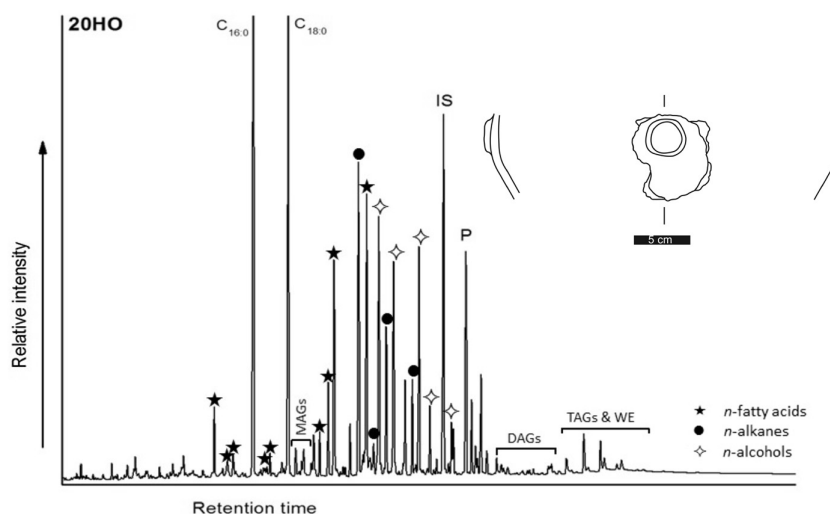


Fig. 7. Partial high-temperature gas chromatogram showing total lipid extracts from pottery sample 20HO from Hočevarica that is characteristic of a mixture of ruminant dairy fat and degraded beeswax.

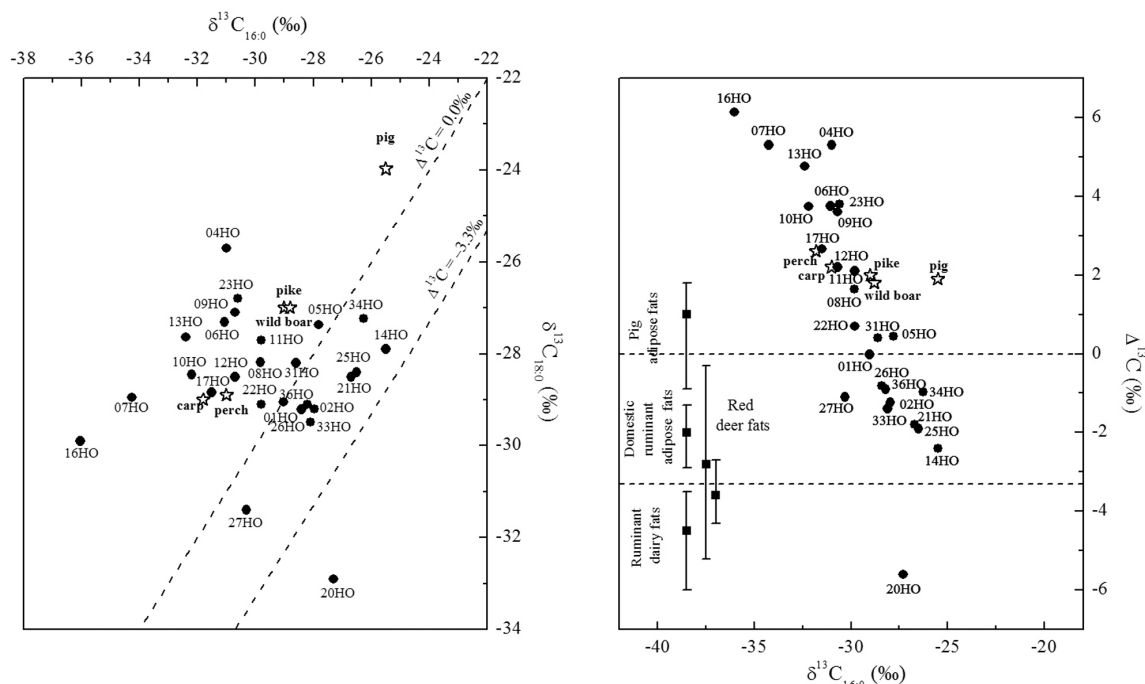


Fig. 8. Plot showing: A) the $\delta^{13}\text{C}_{18:0}$ versus $\delta^{13}\text{C}_{16:0}$ values of some modern reference animal fats and archaeological samples; B) the difference in the $\delta^{13}\text{C}$ values of $\text{C}_{18:0}$ and $\text{C}_{16:0}$ fatty acids ($\Delta^{13}\text{C}$) versus $\delta^{13}\text{C}_{16:0}$ recovered from pottery extracts from Hočevarica. Also shown are the data from modern reference fat: ☆ data from Craig et al. (2007) and the median and ranges of $\delta^{13}\text{C}$ from animals fed exclusively on C_3 diets. The pig adipose fats and ruminant adipose and dairy fats are from Copley et al. (2005), while the wild ruminants are from the UK (Evershed et al. 2002) and red deer from Poland (Craig et al. 2012). All isotope data have been adjusted for the effects of post-industrial carbon (Friedl et al. 1986) in order to compare them with archaeological data.

Only one decorated pot with an appliqué (20HO) indicates the processing of dairy fat. This pot dates to the 2nd settlement phase at Hočevarica and was made with the less common fine-grained fabric with quartz inclusions (Fig. 4; Tab. 2).

The appearance of aquatic biomarkers is associated with nine samples (04HO, 06HO, 09HO, 10HO, 11HO, 12HO, 13HO, 16HO and 17HO), indicating that these vessels were used in the preparation of aquatic resources such as fish and molluscs (Tab. 1). One of the samples with aquatic biomarkers is a pot with an appliqué (11HO; Fig. 4). Most of the samples come from the oldest settlement phase at Hočevarica and have similar technological characteristics in terms of their inclusions (calcite/limestone group), surface and firing treatment. This group of vessels also includes the only samples with a grey-black slip on the surface (10HO).

Moreover, we found that three of the pottery samples (01HO, 22HO and 31HO) were used to process both plant and animal fats. These samples also come from all the settlement phases and are made with calcite/limestone inclusions. Sample 31HO is also a pot with an appliqué and comes from the same con-

text as pot 11HO, which showed the presence of aquatic biomarkers (Fig. 4; Tab.2).

The presence of beeswax in the vessels suggests either the storage of honey or the use of beeswax as a waterproofing agent. Beeswax was detected in five samples (Tab. 2; 20HO, 21HO, 25HO, 26HO, 36HO), of which four come from the 2nd settlement occupation phase and fall into the group with quartz inclusions. As to their morphology, the samples with preserved beeswax include two dishes (21HO, 26HO), one pot that was also used to process dairy fat (20HO), and one bowl (36HO) (Fig. 4). These results suggest that the use of beeswax as a waterproofing agent or the use of honey in the preparation of food was more common in the younger settlement phase at Hočevarica and/or connected to special types of vessels made with a different ceramic fabric.

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